



## PATENT COOPERATION TREATY

## PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Rec'd PCT/PTO 21 DEC 2004

Applicant's or agent's file reference 0000053674		<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/PEA416)	
International application No. PCT/EP 03/06514	International filing date (day/month/year) 20.06.2003	Priority date (day/month/year) 01.07.2002	
International Patent Classification (IPC) or both national classification and IPC C08F20/04			
Applicant BASF AKTIENGESELLSCHAFT et al			
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of 3 sheets.</p>			
<p>3. This report contains indications relating to the following items:</p> <p>I <input checked="" type="checkbox"/> Basis of the opinion</p> <p>II <input type="checkbox"/> Priority</p> <p>III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</p> <p>IV <input type="checkbox"/> Lack of unity of invention</p> <p>V <input checked="" type="checkbox"/> Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p>VI <input type="checkbox"/> Certain documents cited</p> <p>VII <input type="checkbox"/> Certain defects in the international application</p> <p>VIII <input type="checkbox"/> Certain observations on the international application</p>			
Date of submission of the demand  02.12.2003		Date of completion of this report  28.07.2004	
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465		Authorized Officer  Clement, S  Telephone No. +49 89 2399-8512 	

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. **PCT/EP 03/06514**

**1. Basis of the report**

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

**Description, Pages**

1-23 as originally filed

**Claims, Numbers**

1-26 received on 25.06.2004 with letter of 24.06.2004

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).  
☐ the language of publication of the international application (under Rule 48.3(b)).  
☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.  
☐ filed together with the international application in computer readable form.  
☐ furnished subsequently to this Authority in written form.  
☐ furnished subsequently to this Authority in computer readable form.  
☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.  
☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:  
☐ the claims, Nos.:  
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

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**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability;  
citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes: Claims	1-22
	No: Claims	23-26
Inventive step (IS)	Yes: Claims	1-22
	No: Claims	
Industrial applicability (IA)	Yes: Claims	1-26
	No: Claims	

**2. Citations and explanations.**

**see separate sheet**

Ad Section V:

Novelty

DE-A-43 36 299, Example 2, discloses a hydrogel obtained by preparing a monomer starting solution of acrylic acid, a crosslinking monomer, water, glycerol and an initiator and polymerizing said monomer starting solution.

DE'299 does not disclose the addition of sulfur-containing modifying compounds.

Thus, present claims 1 to 22 are novel over the disclosure of DE'299 (Art. 33 (2) PCT).

According to the application, no by-products are contained anymore in the hydrogel obtained by the process according to the application as the modifying agent apparently reacts with the by-products or impurities (see page 12). Therefore, the products *obtained by* the process according to claim 1 neither contain the modifying agent nor the impurities or by-products. That is, the resulting products according to claim 23 and 25 correspond to the hydrogels according to DE'299, in which neither sulfur-containing compounds nor any impurities or by-products are mentioned (0 ppb!).

Moreover, there is a general rule applied that achieving a particularly high level of purity of a known product is not a feature to be regarded as imparting novelty to such a product over the prior art.

Therefore, claims 23 and 24 to 26 are not novel over the disclosure of DE'299 (Art. 33 (2) PCT).

Inventive Step

DE'299 does not suggest to add the selected modifying agent in order to obtain hydrogels of higher purity.

Thus, present claims 1 to 22 involve an inventive step (Art. 33 (3) PCT).

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International application No. PCT/EP 03/06514

Industrial applicability

All claims fulfill the requirements of Art. 33 (4) PCT.

## Claims

1. A process for making a hydrogel comprising 10-90 wt% water, 10-60 wt% of cross-linked hydrophilic polymer made from at least one starting monomer type, and 10-80 wt% of at least one polyol, characterized in that said process comprises the steps of:
- 1) preparing said starting monomer(s) solution from 10-90 wt% water, from 10-60 wt% of said starting monomer(s) and from 10-80 wt% of said polyol(s), and adding a modifying compound in said monomer solution prior to polymerization of the so formed mixture and thereafter
  - 2) polymerizing said monomer(s) within a reaction medium comprising from 10-90 wt% water from 10-60 wt% of said starting monomer(s) and from 10-80 wt% of said polyol(s), in the presence of the modifying compound to thereby form a hydrogel,
- wherein the modifying compound is selected from the group consisting of thiols, sulfites, metabisulfites and bisulfites.
2. A process according to claim 1 wherein the modifying compound is added directly to the monomer solution before the polymerization preferably in a stirring vessel, a tube or a static mixer.
3. A process according to claim 1 or 2 wherein in addition to the modifying compound a scavenger compound is added.
4. A process according to claim 1 - 3 wherein in addition to the modifying compound a chain transfer agent is added.
5. A process according to claim 1 - 4 wherein in addition to the modifying compound a scavenger compound and chain transfer agent is added.
6. A process according to claim 1 - 5 wherein the residual monomer(s) concentration in the hydrogel product of step 1), is below 10000 ppm, preferably below 1000 ppm, more preferably below 500 ppm, even more preferably below 200 ppm, and most preferably below 10 ppm.
7. A process according to claim 1-6 wherein the polymerization of said starting monomer(s) is conducted at a pH 3.5 to 7, preferably 4 to 6.5, more preferably 4.5 to 6.

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8. A process according to claims 1-7 wherein said hydrogel comprises 20-70 wt% water.
- 5 9. A process according to claims 1-8 wherein said adding a modifying compound in step 1) comprises adding to the said monomer premix solution a nucleophile which reacts with said residual starting monomer(s), impurity(s) and/or by-products by an addition reaction.
- 10 10. A process according to claims 1-9 wherein said by-product(s) comprise  $\alpha,\beta$ -unsaturated carbonyl(s) produced from said polyol(s).
11. A process according to claim 10 wherein said polyol is glycerol.
- 15 12. A process according to claims 1-11 wherein said by-product(s) comprise acrolein.
13. A process according to claim 9 wherein the bisulfite is present in amounts of less than 30000 ppm, preferably less than 10000 ppm, more preferably less than 5000 ppm, most preferably less than 1000 ppm, with respect to the product of step 1).
- 20 14. A process according to claims 1-13 wherein the polymerization of said starting monomer(s) is conducted at least partly by UV irradiation.
- 25 15. A process according to claim 1-14 wherein said reaction medium comprises a photoinitiator.
16. A process according to claim 15 wherein said photoinitiator is selected from the group consisting of Darocur 1173, Irgacure 2959, Irgacure 500, and Irgacure 184.
- 30 17. A process according to claim 16 wherein said photoinitiator is used in said reaction medium at a concentration less than 5 wt%, preferably less than 1 wt%, more preferably less than 0.5 wt%, and most preferably less than 0.4 wt%.
- 35 18. A process according to claims 1-17 wherein the polymerization is conducted by UV curing, and the integrated UV intensity at wavelengths less than 280 nm, preferably less than 300 nm, more preferably less than 320 nm is less than 10%, preferably less than 7%, even more preferably less than 4%, most preferably
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less than 1 the total integrated UV intensity with wavelengths less than 400 nm.

- 5 19. A process according to claim 18 wherein said polymerization is carried out under a total UVA energy ranging from 0.1-30 J/cm<sup>2</sup>, preferably from 0.1-25 J/cm<sup>2</sup>, more preferably from 1-20 J/cm<sup>2</sup>.
- 10 20. A process according to claims 1-19 wherein said starting monomer(s) comprise acrylic acid.
21. A process according to claims 1-20 wherein said hydrogel is adhesive.
- 15 22. A process according to claims 1-21 wherein said hydrogel has a tan  $\delta_{25}$  between 0.03 and 3.
23. A hydrogel obtainable by a process of one of the claims 1 to 22.
- 20 24. A hydrogel comprising 10-90 wt% water, 10-60 wt% of cross-linked hydrophilic polymer made from starting monomer(s), and 10-80 wt% of at least one polyol, said hydrogel being prepared by polymerizing said starting monomer(s) in the presence of said water and polyol(s), characterized in that said hydrogel contains less than 100 ppb, preferably less than 50 ppb, and most preferably less than 20 ppb of  $\alpha,\beta$ -unsaturated carbonyl by-product(s) derived from said polyol(s) during polymerization.
- 25 25. A hydrogel according to claim 25 where said  $\alpha,\beta$ -unsaturated carbonyl by-product comprises acrolein.
26. A hydrogel according to claims 23-25 wherein said hydrogel is adhesive.

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